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**APPLICATION FOR LETTERS PATENT**

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**POLISHING SYSTEMS, METHODS OF  
POLISHING SUBSTRATES, AND METHODS OF  
PREPARING LIQUIDS FOR SEMICONDUCTOR  
FABRICATION PROCESSES**

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**INVENTORS**

**Dan G. Custer  
Aaron Trent Ward  
Shawn M. Lewis**

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1 POLISHING SYSTEMS, METHODS OF POLISHING SUBSTRATES,  
2 AND METHODS OF CLEANING POLISHING SLURRY FROM  
3 SUBSTRATE SURFACES

4 TECHNICAL FIELD

5 The invention pertains to methods and apparatuses for increasing  
6 dissolved gas concentrations in liquids and to methods of providing  
7 liquids for semiconductive wafer fabrication processes, such as polishing  
8 systems. The invention also pertains to methods of cleaning polishing  
9 slurry from semiconductive substrate surfaces.

10  
11 BACKGROUND OF THE INVENTION

12 In many semiconductive material fabrication processes it is  
13 desirable to utilize deionized and degassed water. The deionization is  
14 used to remove elemental contaminants from the water and can increase  
15 a resistance of the water to from about 200 kohms to about  
16 1800 kohms.

17 The degassification is used to remove carbon dioxide from the  
18 water. Carbon dioxide can influence a pH of the water. The  
19 degassification also, however, removes other gasses from water besides  
20 carbon dioxide. Such other gasses can include, for example, oxygen and  
21 nitrogen. An example unit for degassifying water is a Liquicell unit  
22 (available from Hoechst Celanese Corp. at 13800 South Lake Drive,  
23 Charlotte, N.C. 28273), which removes gasses via a gas permeable  
24 membrane.

1       The deionization and degassification of water is typically done on  
2 a system-wide scale in a semiconductive material fabrication plant.  
3 Accordingly, all water supplied to the various fabrication units of the  
4 plant is degassed and deionized.  
5

## 6       SUMMARY OF THE INVENTION

7       The invention encompasses methods and apparatuses for increasing  
8 dissolved gas concentrations in liquids, and methods of providing liquids  
9 for semiconductive wafer fabrication processes, such as polishing systems.  
10 The invention also encompasses polishing systems for polishing  
11 semiconductive material substrates, and methods of cleaning polishing  
12 slurry from semiconductive substrate surfaces.

13       In one aspect, the invention encompasses a method of preparing  
14 a liquid for a semiconductor fabrication process. A liquid is provided,  
15 and a gas is injected into the liquid to increase a total dissolved gas  
16 concentration in the liquid.

17       In another aspect, the invention encompasses a method of cleaning  
18 a polishing slurry from a substrate surface. A substrate surface is  
19 provided, and a polishing slurry is provided in contact with the substrate  
20 surface. A liquid is provided. A gas is injected into the liquid to  
21 increase a total dissolved gas concentration in the liquid. After the  
22 injecting, the liquid is provided against the substrate surface to displace  
23 the polishing slurry from the substrate surface.  
24

1 In yet another aspect, the invention encompasses a method of  
2 polishing a substrate surface. A polishing slurry is provided between  
3 a substrate surface and a polishing pad. The substrate surface is  
4 polished with the polishing slurry. The polishing slurry is removed from  
5 the substrate surface. The removing comprises the following. A liquid  
6 is provided. A first gas is removed from the liquid to reduce a total  
7 dissolved gas concentration in the liquid. After removing the first gas,  
8 a second gas is dissolved in the liquid to increase the total dissolved  
9 gas concentration in the liquid. After dissolving the second gas, the  
10 liquid is provided between the substrate surface and the polishing pad  
11 to displace the polishing slurry from the substrate surface.

### 12 BRIEF DESCRIPTION OF THE DRAWINGS

14 Preferred embodiments of the invention are described below with  
15 reference to the following accompanying drawings.

16 Fig. 1 is a fragmentary, diagrammatic cross-sectional view of a  
17 polishing apparatus for polishing a semiconductive wafer.

18 Fig. 2 is a top view of the Fig. 1 apparatus.

19 Fig. 3 is a diagrammatic and schematic cross-sectional view of a  
20 gassification apparatus of the present invention.

## DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

This disclosure of the invention is submitted in furtherance of the constitutional purposes of the U.S. Patent Laws "to promote the progress of science and useful arts" (Article 1, Section 8).

In accordance with the present invention it is recognized that liquids utilized for various wafer fabrication processes will preferably have at least a threshold dissolved gas concentration. It has been discovered that if water utilized in polishing processes has a dissolved gas concentration below a threshold, wafers will slip out of a polishing apparatus at a significantly higher frequency than if the dissolved gas concentration is above the threshold. It is also expected that if water utilized in a semiconductor wafer etch or polish processes has a dissolved gas concentration below a threshold, the water will become a better solvent for various etchant or polishing compounds than if the dissolved gas concentration is above the threshold. The better solvent properties of the water can alter an etch or polish rate and lead to defects in the etched or polished wafer. Such defects can include domed regions, inclusions, and cavities. Accordingly, the present invention encompasses methods of providing dissolved gasses in water and other liquids.

An example polishing process is described with reference to a polishing apparatus 10 in Figs. 1 and 2. Polishing apparatus 10 can, for example, be an apparatus configured to accomplish chemical-

1 mechanical polishing. Apparatus 10 comprises a polishing pad 12 and  
2 semiconductive wafer holders 14 and 16.

3 Wafer holders 14 and 16 hold a pair of semiconductive wafers 18  
4 and 20 adjacent a surface of the polishing pad 12. Wafer holders 14  
5 and 16 comprise sidewalls 22 and 24, respectively. Generally,  
6 semiconductive wafers 18 and 20 are circular in shape, and sidewalls 22  
7 and 24 are circular and ring-shaped to completely encircle wafers 18  
8 and 20.

9 In operation, a polishing slurry is provided between semiconductive  
10 wafers 18 and 20, and polishing pad 12. The polishing slurry can  
11 comprise, for example, ILD 1300 or MSW 1300 manufactured by Rodel,  
12 Inc. of Delaware. After the slurry is provided, wafer holders 14  
13 and 16 are utilized to move wafers 18 and 20 relative to polishing  
14 pad 12 to polish surfaces of wafers 18 and 20 with the slurry.

15 As shown in Fig. 2, wafer holders 16 and 18 are preferably  
16 configured to move semiconductive wafers 18 and 20 in a number of  
17 directions relative to polishing pad 12 during a polishing process. Such  
18 directions are illustrated by axes "A," "B," "C," "D," and "E." Axes A,  
19 B, and E are rotational axes, and axes C and D are translational axes.  
20 The many varied rotations and translations illustrated in Fig. 2 enable  
21 wafers 18 and 20 to be polished quickly and uniformly.

22 Polishing apparatus 10 comprises a pair of nozzles 27. After a  
23 surface of wafers 18 and 20 is polished, a liquid is introduced through  
24 nozzles 27 and onto polishing pad 12 to displace the polishing slurry

1 from between wafers 18 and 20 and polishing pad 12. Wafers 18  
2 and 20 typically are moved relative to polishing pad 12 as the liquid  
3 is provided onto polishing pad 12. The liquid preferably comprises  
4 deionized water, and more preferably consists essentially of deionized  
5 water having some dissolved gas therein. In accordance with the  
6 present invention, it has been discovered that if the liquid comprises too  
7 low of a dissolved gas concentration, excess friction will develop between  
8 wafers 18 and 20 and polishing pad 12. Such excess friction can result  
9 in wafers 18 and 20 being disastrously expelled from wafer holders 14  
10 and 16, a so-called "slip-out" of the wafers.

11 A method for determining total dissolved gas in water is to  
12 measure the concentration of dissolved oxygen. As discussed in the  
13 Background section of this disclosure, degassification procedures are  
14 generally not selective for particular dissolved gasses and lower all  
15 dissolved gasses in a liquid. A dissolved oxygen concentration can be  
16 particularly conveniently measured by methods known to persons of  
17 ordinary skill in the art. It is therefore expedient to quantitate a  
18 dissolved oxygen concentration and to use this as an indicator of a total  
19 dissolved gas concentration in a source of water. It has been found  
20 experimentally that if the dissolved oxygen concentration in a source of  
21 water is above about 150 parts per billion (ppb), preferably above  
22 about 190 ppb, and more preferably above about 200 ppb, slip-out of  
23 wafers can be avoided. However, when the dissolved oxygen  
24 concentration falls to below 150 ppb slip-out becomes unacceptably

1 frequent. Often, slip-out becomes unacceptably frequent if the dissolved  
2 oxygen concentration falls to below 200 ppb. Currently utilized  
3 degassification procedures will reduce dissolved oxygen concentrations to  
4 about 4 ppb, which is too low for many polishing processes.  
5 Accordingly, it is desirable to regassify water prior to utilization in  
6 polishing processes.

7 The gas provided in a liquid during a regassification procedure  
8 can have a composition different from the gas removed from the liquid  
9 during a degassification procedure. The gas removed from the liquid  
10 during the degassification process is a first gas which will generally have  
11 a composition similar to that of the atmosphere. The gas provided  
12 back into the liquid during a regassification is a second gas which is  
13 preferably a relatively cheap and non-reactive gas, such as argon or  
14 nitrogen. The second gas is preferably provided to a concentration of  
15 at least 200 ppb, preferably of from about 450 ppb to about 550 ppb,  
16 and more preferably of at least about 500 ppb. Such concentration of  
17 second gas has been found experimentally to convert a degassified liquid  
18 having 4 ppb of dissolved oxygen to a liquid which will significantly  
19 reduce slip-out of wafers. An exemplary upper limit of the second gas  
20 which can be added to deionized water is about 7 parts per million  
21 (ppm), as this is about the maximum amount of dissolved gas that  
22 deionized water can retain at room temperature and atmospheric  
23 pressure.  
24



1 A preferred method for regassifying a liquid is described with  
2 reference to a regassification apparatus 50 in Fig. 3. Apparatus 50  
3 comprises a pipe 52 through which a liquid flows from a source 54 to  
4 a polishing apparatus 56. Pipe 52 can comprise, for example, a  
5 nominal half-inch inner diameter. Pipe 52 comprises a tee 58 wherein  
6 a gas is injected with the liquid to increase a dissolved gas  
7 concentration in the liquid. The gas flows from a source 60, through  
8 a pressure regulator 62, a flowmeter 64, a pressure/flow switch 66, a  
9 check valve 68, and a gas dispersion unit 70 to inject with liquid in  
10 tee 58. Source 60 preferably comprises the gas stored at pressure  
11 greater than atmospheric pressure.

12 Gas dispersion unit 70 can comprise, for example, a sintered  
13 filter. A sintered filter 70 can comprise a number of materials and  
14 constructions known to persons of skill in the art. For example,  
15 filter 70 can comprise a stainless steel filter having about 0.5 micron  
16 pores. Filter 70 comprises a nipple 72 extending beneath tee 58 and  
17 having, for example, about a one-quarter inch diameter.

18 In an example process wherein nitrogen is flowed into water, a  
19 pressure of the nitrogen will preferably be maintained at about 100  
20 pounds per square inch gauge (psig), and a flow of the nitrogen will  
21 preferably be maintained at about 750 cubic centimeters per minute  
22 (ccpm). Also, check valve 68 will preferably be set to a pressure of 2  
23 psi. The water will preferably be flowed through pipe 52 at a rate of  
24

1 from about 2.5 gallons per minute to about 4 gallons per minute, and  
2 a pressure of 45-50 psig.

3 Pipe 52 defines a tube through which fluid flows. The liquid  
4 from source 54 and gas from source 60 meet within such tube. By  
5 having the liquid confined in a tube as it is injected with gas, a  
6 controlled pressure of liquid and gas can be maintained to substantially  
7 ensure that the gas dissolves within the liquid.

8 The apparatus of Fig. 3 represents a preferred method for  
9 increasing a total dissolved gas concentration in a liquid. Another  
10 method for increasing a total dissolved gas concentration in a liquid is  
11 to introduce a flush gas in a gas-permeable-membrane-based  
12 degassification procedure. An example gas-permeable-membrane-based  
13 degassification procedure is a Liquicell procedure. The flush gas is  
14 provided at the membrane during degassification and helps to remove  
15 inherent gasses from a liquid as the liquid is degassified. Some of the  
16 flush gas will remain in the liquid after the liquid passes through the  
17 degassification apparatus. For instance, if nitrogen is utilized as a flush  
18 gas in a degassification membrane procedure, the nitrogen will essentially  
19 replace at least some of the carbon dioxide and other gasses originally  
20 present in the liquid. Thus, the water is both degassed and regassified  
21 in a common step.

22 Persons of ordinary skill in the art will recognize that a dissolved  
23 nitrogen concentration in the "degassed" water can be adjusted by  
24 adjusting a flow of the nitrogen flush gas. If the water is to be

1 utilized in a polishing process of the present invention, the nitrogen gas  
2 flow rate will preferably be adjusted to result in nitrogen being present  
3 in the water at concentrations in excess of 200 ppb, and more  
4 preferably at concentrations in a range of from 450 ppb to about 550  
5 ppb.

6 The methods discussed above for regassifying liquids have been  
7 described for applications in which the regassified liquids are utilized to  
8 displace slurries from polishing apparatuses. It is to be understood that  
9 such regassified liquids can also be utilized for other semiconductive  
10 wafer fabrication processes. For instance, the regassified liquids could  
11 be utilized for cleaning semiconductive wafers prior to processing steps.  
12 For example, semiconductive wafers are frequently washed with deionized  
13 water prior to polishing of the wafers in a polishing apparatus. Such  
14 deionized water can be regassified water produced in accordance with  
15 methods of the present invention.

16 In compliance with the statute, the invention has been described  
17 in language more or less specific as to structural and methodical  
18 features. It is to be understood, however, that the invention is not  
19 limited to the specific features shown and described, since the means  
20 herein disclosed comprise preferred forms of putting the invention into  
21 effect. The invention is, therefore, claimed in any of its forms or  
22 modifications within the proper scope of the appended claims  
23 appropriately interpreted in accordance with the doctrine of equivalents.  
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